

# **DATA VALIDATION REPORT**

Bunker Hill Central Treatment Plant Kellogg, Idaho

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# **ACRONYMS**

% percent

CCB continuing calibration blank

CCV continuing calibration verification

CLP Contract Laboratory Program

COC chain of custody

EPA United States Environmental Protection Agency

ICAL initial calibration

ICV initial calibration verification

ID identification

LCS laboratory control sample

LCSD laboratory control sample duplicate

MDL method detection limit

mg/L milligrams per liter

MS matrix spike

MSD matrix spike duplicate

QC quality control

RL reporting limit

RPD relative percent difference

SAP sampling and analysis plan

SM Standard Method

SVL SVL Analytical Inc.

TSS total suspended solids

# **DATA VALIDATION REPORT**

# Bunker Hill Central Treatment Plant Kellogg, Idaho

#### 1.0 INTRODUCTION

Amec Foster Wheeler Environment & Infrastructure, Inc. (Amec Foster Wheeler) collected 24 water samples (including 3 field duplicates, a field blank and an equipment blank) between January 1 and 31, 2018 from the Bunker Hill Central Treatment Plant in Kellogg, Idaho. Amec Foster Wheeler submitted the samples to SVL Analytical Inc. (SVL), located in Coeur D'Alene, Idaho, where they were assigned to sample delivery groups X8A0001, X8A0002, X8A0026, X8A0069, X8A0092, X8A0093, X8A0129, X8A0201, X8A0219, X8A0247, X8A0308, X8A0333, X8A0357, X8A0406, X8A0427, X8A0456. SVL analyzed the samples for total metals by United States Environmental Protection Agency (EPA) Method 200.7, total suspended solids (TSS) by Standard Methods for the Examination of Water and Wastewater (SM) 2540D; and pH by SM 4500-H B. A list of these samples by field sample identification (ID), sample collection date, and the laboratory sample IDs is presented in Table 1.

# 2.0 DATA VALIDATION METHODOLOGY

Amec Foster Wheeler performed Stage 4 validation on samples KT-01-01-18, QC-01-01-18, and KT-01-04-18. The Stage 4 validation includes review and recalculation of the laboratory's analytical report and the raw analytical data. The remainder of the data underwent EPA Stage 2B validation, which includes review of sample- and instrument-specific quality control (QC) samples on data summary forms, but does not include review or validation of the raw analytical data. This data validation has been performed in general accordance with:

- Amec Foster Wheeler, 2017. Operations & Maintenance Services Sampling and Analysis Plan (SAP), Bunker Hill Central Treatment Plant Upgrade Project, Kellogg, Idaho, March 2017.
- EPA, 2014. EPA Contract Laboratory Program (CLP) National Functional Guidelines for Inorganic Superfund Data Review, EPA-540 R 013 001.
- The analytical methods referenced by the laboratory.

The laboratory's certified analytical report and supporting documentation were reviewed to assess the following:

- Data package and electronic data deliverable completeness;
- Chain-of-custody (COC) compliance;
- Sample Receipt;
- Holding time compliance;
- Initial calibration (ICAL), initial calibration verification (ICV), and continuing calibration verification (CCV) compliance with method specified criteria;
- Presence or absence of laboratory contamination as demonstrated by calibration and laboratory blanks;
- Accuracy and bias as demonstrated by recovery of surrogate spikes, laboratory control sample (LCS), and matrix spike (MS) samples;
- Analytical precision as relative percent difference (RPD) of analyte concentration between laboratory duplicates, LCSs/LCS duplicates (LCSDs), or MS/MS duplicates (MSDs);
- Sampling and analytical precision as RPD of analyte concentration between field duplicates;
- Internal standard and surrogate compound recoveries:
- Analyte identification and quantification verification from raw analytical data (Stage 4 validation only); and
- Insofar as possible, the degree of conformance to method requirements and good laboratory practices

# 3.0 EXPLANATION OF DATA QUALITY INDICATORS

Summary explanations of the specific data quality indicators reviewed during data validation are presented below.

# 3.1 LABORATORY CONTROL SAMPLE RECOVERIES

LCSs are aliquots of analyte free matrices that are spiked with the analytes of interest for an analytical method, or a representative subset of those analytes. The spiked matrix is then processed through the same analytical procedures as the samples they accompany. LCS recovery is an indication of a laboratory's ability to successfully perform an analytical method in an interference free matrix.

# 3.2 MATRIX SPIKE RECOVERIES

MSs and MSDs are prepared by adding known amounts of the analytes of interest for an analytical method, or a representative subset of those analytes, to an aliquot of sample. The spiked sample is then processed through the same extraction, concentration, cleanup, and analytical procedures as the unspiked samples in an analytical batch.

MS recovery and precision are an indication of a laboratory's ability to successfully recover an analyte in the matrix of a specific sample or closely related sample matrices. It is important not to apply MS results for any specific sample to other samples without understanding how the sample matrices are related.

# 3.3 BLANK CONCENTRATIONS

Blank samples are aliquots of analyte free matrix that are used as negative controls to verify that the sample collection, storage, preparation, and analysis system does not produce false positive results.

Laboratory blanks are processed by the laboratory using exactly the same procedures as the field samples. Target analytes should not be found in laboratory blanks.

Target analytes should not be found in laboratory blanks. When target analytes are detected in blanks, analyte concentrations in associated samples less than five times the concentration detected in the blank will be U qualified as being not detected.

# 3.4 LABORATORY DUPLICATES

Laboratory duplicate analysis verifies acceptable method precision by the laboratory at the time of preparation and analysis and/or sampling precision at the time of collection.

# 4.0 DEFINITIONS OF QUALIFIERS THAT MAY BE ADDED DURING DATA VALIDATION

- **J** The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- R The sample result is rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

- **U** The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
- **UJ** The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

# 5.0 QUALIFICATION REASON CODES

The following reason codes were applied to the data during data validation:

- DL The analyte concentration is between the method detection limit (MDL) and the reporting limit (RL).
- HD High laboratory duplicate RPD
- HT Holding time exceeded.

# 6.0 CHAIN OF CUSTODY AND SAMPLE RECEIPT CONDITION DOCUMENTATION

The samples were received at the laboratory under proper COC, intact, properly preserved, and at temperatures less than the SAP-specified maximum of 6 degrees Celsius.

# 7.0 SPECIFIC DATA VALIDATION FINDINGS

Results from these samples may be considered usable with the limitations and exceptions described in Sections 7.1 through 8.0. Qualifiers added during validation are summarized in Table 2.

# 7.1 METALS BY EPA METHOD 200.7

Total metals results generated by SVL may be considered usable with the limitations described in Sections 7.1.1 through 7.1.9.

# 7.1.1 Holding Times

All samples were analyzed for metals within the SAP-specified holding of 180 days

# 7.1.2 Initial and Continuing Calibration Verification

ICV and CCV recoveries were within method-specified limits.

# 7.1.3 Initial and Continuing Calibration Blanks

Target analytes were not detected in the initial calibration blanks and continuing calibration blanks (CCBs), with the following exceptions.

Zinc was detected at a concentration of 0.003 milligrams per liter (mg/L) in the CCB associated with the analysis of samples KT-01-01-18, QC-01-01-18, and KT-01-04-18. Zinc was detected in the associated samples at concentrations greater than five times the concentration detected in the blank and data usability is not adversely affected by the blank detection.

# 7.1.4 Laboratory, Equipment, and Trip Blanks

Target analytes were not detected in the laboratory, equipment, and trip blanks associated with the analysis of these samples, with the following exception.

Zinc was detected at a concentration of 0.005 mg/L in the laboratory blank associated with the analysis of sample 006-01-19-18. Zinc was detected in the associated sample at a concentration greater than five times the concentration detected in the blank and data usability is not adversely affected by the blank detection.

# 7.1.5 Laboratory Control Sample Accuracy and Precision

LCS and LCSD recoveries were within the SAP-specified limits, and RPDs between the LCS and LCSD results were less than the SAP-specified maximum of 20 percent (%).

# 7.1.6 Laboratory Duplicates

SVL performed duplicate analyses on project samples KT-12-25-17, KT-01-01-18, 006-01-01-18, 006-01-03-18, 006-01-05-18, 006-01-08-18, PTM-01-11-18, 006-01-10-18, 006-01-12-18, 006-01-15-18, KT-01-15-18, 006-01-17-18, 006-01-19-18, 006-01-22-18, 006-01-24-18, 006-01-26-18, 006-01-29-18, and 006-01-31-18. RPDs between source and duplicate results were less than the SAP-specified maximum of 20%, with the following exception:

The RPD for zinc was high at 24.8% in the duplicate analysis of sample 006-01-05-18. Amec Foster Wheeler J qualified the detected zinc result from sample 006-01-05-18 due to potential analytical imprecision. (J-HD)

# 7.1.7 Matrix Spikes/Matrix Spike Duplicates

SVL performed MS and MSD analyses on project samples KT-12-25-1, PTM-01-11-187, KT-01-01-18, 006-01-01-18, 006-01-03-18, 006-01-05-18, 006-01-08-18, 006-01-10-18, 006-01-12-18, 006-01-15-18, KT-01-15-18, 006-01-17-18, 006-01-19-18, 006-01-22-18, 006-01-24-18, 006-01-26-18, 006-01-29-18, and 006-01-31-18 for total metals. MS/MSD recoveries were within SAP-specified limits and RPDs between MS and MSD results were less than the laboratory-specified maximum of 20%, with the following exceptions:

- Manganese and zinc recoveries were low at 5.20% and -93.5%, respectively, in the MS performed on sample KT-01-01-18. Manganese and zinc were detected in the native unspiked sample at concentrations greater than four times the spike concentrations and data usability cannot be evaluated based on the performance of these analytes in this sample.
- Manganese (18.4%, 56.6%) and zinc (20.0%, MSD) recoveries were low in the MS and MSD performed on sample KT-12-25-17. Manganese and zinc were detected in the native unspiked sample at concentrations greater than four times the spike concentrations and data usability cannot be evaluated based on the performance of these analytes in this sample.
- Manganese (45.5%, 20.8%) and zinc (-23.4%, -140%) recoveries were low in the MS and MSD performed on sample KT-01-15-18. Manganese and zinc were detected in the native unspiked sample at concentrations greater than four times the spike concentrations and data usability cannot be evaluated based on the performance of these analytes in this sample.
- Manganese recoveries were outside SAP-specified limits in samples 006-01-01-18 (139%, MS), 006-01-05-18 (69.0%, MSD), 006-01-08-18 (39.9%, -35.8%), 006-01-10-18 (-21.9%, MSD), 006-01-12-18 (40.6%, 140%), 006-01-15-18 (134%, MSD), 006-01-24-18 (237%, -28.5%), 006-01-26-18 (53.1%, MS), 006-01-29-18 (381%, 446%), and 006-01-31-18 (394%, 247%). Manganese as detected in the native unspiked samples at concentrations greater than four times the spike concentrations and data usability cannot be evaluated based on the performance of this analyte in these samples.

# 7.1.8 Analytical Sensitivity

Amec Foster Wheeler compared RLs for cadmium, lead, manganese, and zinc against applicable discharge limits to confirm that the RLs were sufficiently low to meet the discharge limits. Non-detect results were reported to RLs less than the applicable discharge limits.

# 7.1.9 Data Reporting and Analytical Procedures

SVL J qualified analytes with concentrations between the MDL and the RL. Amec Foster Wheeler agrees that these results are quantitatively uncertain and has maintained SVL's J qualifiers. (J-DL)

# 7.2 TOTAL SUSPENDED SOLIDS BY SM 2540D

TSS results generated by SVL may be considered usable with the limitations described in Sections 7.2.1 through 7.2.6.

# 7.2.1 Holding Times

All samples were analyzed for TSS within the SAP-specified holding time of 7 days.

# 7.2.2 Laboratory Blanks

TSS was not detected in the laboratory blanks associated with the analysis of these samples.

# 7.2.3 Laboratory Control Sample Accuracy and Precision

LCS and LCSD recoveries were within the laboratory-specified 90 to 110% limits and RPDs between the LCS and LCSD results were less than the laboratory-specified maximum of 10%.

# 7.2.4 Laboratory Duplicates

SVL performed duplicate analyses on project samples KT-12-25-17, KT-01-01-18, 006-01-01-18, 006-01-03-18, 006-01-05-18, 006-01-08-18, 006-01-10-18, 006-01-12-18, 006-01-15-18, KT-01-15-18, 006-01-17-18, 006-01-19-18, 006-01-22-18, 006-01-24-18, 006-01-26-18, 006-01-29-18, 006-01-31-18. RPDs between source and duplicate results were less than laboratory-specified maximum of 10%, with the following exceptions:

RPDs for TSS were high in the duplicate analyses performed on samples 006-01-01-18 and 006-01-19-18 at 66.7% and 100%, respectively. The differences between primary and duplicate results were less than the RL, demonstrating acceptable analytical precision.

# 7.2.5 Analytical sensitivity

Amec Foster Wheeler compared RLs for TSS against applicable discharge limits to confirm that the RLs were sufficiently low to meet the discharge limits. Non-detect results were reported to RLs less than the applicable discharge limits.

# 7.2.6 Data Reporting and Analytical Procedures

SVL J qualified analytes with concentrations between the MDL and the RL. Amec Foster Wheeler agrees that these results are quantitatively uncertain and has maintained SVL's J qualifiers. (J-DL)

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# 7.3 PH BY SM 4500B

pH results generated by SVL may be considered usable with the limitations described in Sections 7.3.1 through 7.3.4.

# 7.3.1 Holding Times

All samples were analyzed for pH after the method-specified 15-minute hold time had passed. Amec Foster Wheeler J qualified the pH results from these samples because of the missed hold time. (J-HT)

# 7.3.2 Laboratory Control Sample Accuracy

LCS recoveries were within the laboratory-specified 98.5 to 101.5% limits.

# 7.3.3 Laboratory Duplicates

SVL performed duplicate analyses on samples KT-12-25-17, KT-01-01-18, 006-01-01-18, 006-01-03-18, 006-01-05-18, 006-01-08-18, 006-01-10-18, 006-01-12-18, 006-01-15-18, KT-01-15-18, 006-01-17-18, 006-01-19-18, 006-01-22-18, 006-01-24-18, 006-01-26-18, 006-01-29-18, and 006-01-31-18. RPDs between source and duplicate results were less than laboratory-specified maximum of 5%.

# 7.3.4 Data Reporting and Analytical Procedures

There were no data anomalies associated with the pH analysis of these samples.

# 8.0 FIELD DUPLICATES

Field duplicates were collected with samples:

- KT-01-01-18 (QC-01-01-18),
- PTM-01-11-18 (QC-01-11-18), and
- . 006-01-26-18 (QC-01-26-18).

Target analyte detections are summarized in Table 3. Precision values were less than the QAPP-specified maximum of 30%, or the differences between detected concentrations were less than the RL, demonstrating acceptable sampling and analytical precision.

# 9.0 SUMMARY AND CONCLUSIONS

Amec Foster Wheeler reviewed 130 data records from field samples during this validation. All the data generated are usable and of acceptable quality with the addition of qualifiers presented in Table 2. Qualifier definitions are summarized in Section 4.0, reason codes are summarized in Section 5.0, and qualified data are summarized below.

Amec Foster Wheeler J qualified 32 records (24.8%) as being estimated concentrations because of hold time exceedances high duplicate analysis RPD, or analyte concentrations between the MDL and RL.

No records were rejected and 100% of the data should be considered valid with the addition of the qualifiers presented in Table 2.

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# **REFERENCES**

Amec Foster Wheeler, 2017. Operations & Maintenance Services Sampling and Analysis Plan (SAP), Bunker Hill Central Treatment Plant Upgrade Project, Kellogg, Idaho, March 2017.

EPA, 2016. EPA Contract Laboratory Program (CLP) National Functional Guidelines for Inorganic Superfund Data Review, EPA-540-R-2016-001.

# **LIMITATIONS**

This report was prepared exclusively for the Bunker Hill Central Treatment Plant by Amec Foster Wheeler Environment & Infrastructure, Inc. The quality of information, conclusions, and estimates contained herein is consistent with the level of effort involved in Amec Foster Wheeler services and based on: i) information available at the time of preparation, ii) data supplied by outside sources, and iii) the assumptions, conditions, and qualifications set forth in this report. This data validation report is intended to be used by the Bunker Hill Central Treatment Plant in Kellogg, Idaho only, subject to the terms and conditions of its contract with Amec Foster Wheeler. Any other use of, or reliance on, this report by any third party is at that party's sole risk.

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**TABLES** 

# TABLE 1 Field Samples Submitted to SVL Analytical, Inc. Bunker Hill Central Treatment Plant Upgrade Program Kellogg, Idaho

Field	Collection SVL Analytical, Inc.		N-4		
Sample ID	Date	Sample ID	Notes		
006-01-01-18	1/1/2018	X8A0001-01			
KT-01-01-18	1/1/2018	X8A0002-01	Stage 4 Validation		
QC-01-01-18	1/1/2018	X8A0002-02	Stage 4 Validation, Field Duplicate of KT-01-01-18		
KT-01-04-18	1/4/2018	X8A0002-03	Stage 4 Validation		
006-01-03-18	1/3/2018	X8A0026-01			
006-01-05-18	1/5/2018	X8A0069-01			
006-01-08-18	1/8/2018	X8A0092-01			
KT-01-08-18	1/8/2018	X8A0093-01			
KT-01-11-18	1/11/2018	X8A0093-02			
PTM-01-11-18	1/11/2018	X8A0093-03			
QC-01-11-18	1/11/2018	X8A0093-04	Field Duplicate of PTM-01-11-18		
TB-01-11-18	1/11/2018	X8A0093-05	Trip Blank		
RB-01-11-18	1/11/2018	X8A0093-06	Equipment Blank		
006-01-10-18	1/10/2018	X8A0129-01			
006-01-12-18	1/12/2018	X8A0201-01			
006-01-15-18	1/15/2018	X8A0219-01			
006-01-17-18	1/17/2018	X8A0247-01			
006-01-19-18	1/19/2018	X8A0308-01			
006-01-22-18	1/22/2018	X8A0333-01			
006-01-24-18	1/24/2018	X8A0357-01			
006-01-26-18	1/26/2018	X8A0406-01			
QC-01-26-18	1/26/2018	X8A0406-02	Field Duplicate of 006-01-26-18		
006-01-29-18	1/29/2018	X8A0427-01			
006-01-31-18	1/31/2018	X8A0456-01			

#### Notes:

ID = identification

# TABLE 2 Qualifiers Added During Data Validation Bunker Hill Central Treatment Plant Upgrade Program Kellogg, Idaho

Sample	Methods	Analytes	Concentrations	Qualifiers and	
IDs	Wicthods	Allalytos	Concentiations	Reason Codes	
006-01-01-18	2540D	TSS	0.400 mg/L	J DL	
006-01-01-18	4500H	рН	7.00 SU	J HT	
006-01-03-18	2540D	TSS	0.800 mg/L	J DL	
006-01-03-18	4500H	рН	7.00 SU	J HT	
006-01-05-18	2540D	TSS	0.200 mg/L	J DL	
006-01-05-18	4500H	рН	7.00 SU	J HT	
006-01-05-18	EPA 200.7	Zinc	0.403 mg/L	J HD, DL	
006-01-08-18	4500H	рН	7.00 SU	J HT	
006-01-10-18	4500H	рН	6.90 SU	J HT	
006-01-12-18	4500H	рН	7.10 SU	J HT	
006-01-15-18	2540D	TSS	0.600 mg/L	J DL	
006-01-15-18	4500H	рН	7.10 SU	J HT	
006-01-17-18	2540D	TSS	0.400 mg/L	J DL	
006-01-17-18	4500H	рН	7.20 SU	J HT	
006-01-19-18	2540D	TSS	0.600 mg/L	J DL	
006-01-19-18	4500H	рН	7.00 SU	J HT	
006-01-22-18	2540D	TSS	0.600 mg/L	J DL	
006-01-22-18	4500H	рН	7.00 SU	J HT	
006-01-24-18	4500H	рН	7.10 SU	J HT	
006-01-26-18	4500H	рН	7.00 SU	J HT	
006-01-26-18	EPA 200.7	Cadmium	0.00180 mg/L	J DL	
006-01-29-18	2540D	TSS	0.800 mg/L	J DL	
006-01-29-18	4500H	рН	7.10 SU	J HT	
006-01-31-18	4500H	рН	7.10 SU	J HT	
KT-01-01-18	4500H	рН	2.90 SU	J HT	
KT-01-04-18	4500H	pН	2.90 SU	J HT	
KT-01-08-18	4500H	pН	3.30 SU	J HT	
KT-01-11-18	4500H	pН	3.00 SU	J HT	
PTM-01-11-18	4500H	pН	7.30 SU	J HT	
QC-01-01-18	4500H	pН	2.90 SU	J HT	
QC-01-11-18	4500H	pH	7.40 SU	J HT	
QC-01-26-18	4500H	рН	6.90 SU	J HT	

# Notes:

SU = Standard Units

TSS = Total Suspended Solids

mg/L = milligrams per liter

EPA = United States Environmental Protection Agency

#### **Qualifier Definition:**

J = The analyte was positively identified; the associated numerical value is approximate.

# **Reason Codes:**

DL = The analyte concentration is between the method detection limit and the reporting limit.

HD = high duplicate RPD

# TABLE 3 Field Duplicate Detections Bunker Hill Central Treatment Plant Upgrade Program Kellogg, Idaho

Analyte	Method	Average RL	Primary Concentration	Field Duplicate	RPD	Notes			
KT-01-01-18 and QC-01-01-18									
TSS	2540D	5.0 mg/L	34	34	0.0%				
рН	4500H	SU	2.9 J	2.9 J	0.0%				
Cadmium	EPA 200.7	0.00200 mg/L	0.147	0.153	4.0%				
Lead	EPA 200.7	0.00750 mg/L	0.491	0.510	3.8%				
Manganese	EPA 200.7	0.00800 mg/L	28.2	29.1	3.1%				
Zinc	EPA 200.7	0.100 mg/L	76.5	76.2	0.4%				
006-01-26-18 and QC-01-26-18									
TSS	2540D	5.0 mg/L	1.4	1.4	0.0%				
рН	4500H	SU	7.0 J	6.9 J	1.4%				
Cadmium	EPA 200.7	0.00200 mg/L	0.00180 J	0.00200	10.5%				
Manganese	EPA 200.7	0.00800 mg/L	40.0	40.8	2.0%				
Zinc	EPA 200.7	0.100 mg/L	0.457	0.475	3.9%				
PTM-01-11-18 and QC-01-11-18									
TSS	2540D	5.0 mg/L	1.2	1.6	28.6%				
рН	4500H	SU	7.3 J	7.4 J	1.4%				
Cadmium	EPA 200.7	0.00200 mg/L	1.11	1.12	0.9%				
Lead	EPA 200.7	0.00750 mg/L	0.0544	0.0550	1.1%				
Zinc	EPA 200.7	0.100 mg/L	10.6	10.8	1.9%				

#### Notes:

mg/L = milligrams per liter

TSS = Total Suspended Solids

RPD = relative percent difference

SU = standard units

RL = reporting limit

EPA = United States Environmental Protection Agency

# **Qualifier Definition:**

J = The analyte was positively identified; the associated numerical value is approximate.